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June 25, 1959

Dear Sir:

This letter report describes the research performed under Task Order No. C during the month of May, 1959.

During this period, six runs, namely, 12, 13, 14, 14-A, 4-A, and 4-B, were made and the data from Run 11, reported last month, were processed.

Runs 12, 13, and 14 were 1/10-scale tests. The quantity of catalyst used in each test was the amount contained in one-half gallon of saturated solution at the initial test temperature. The total volume of water used in Run 12 and in the previous 1/10-scale tests was 56 gallons. In Runs 13 and 14, the volume of water was reduced to 40 and 25 gallons, respectively. The experimental data for these three runs, as well as the data for Run 11, reported previously, are shown on the following page.

Run 14, which involved 25 gallons of water, 10 pounds of sodium borohydride, and a catalyst solution saturated at 33 F, was completed in 10 minutes. This is much faster than expected by comparison with previous Runs 10 and 13, which used 56 and 40 gallons of water, respectively. Also it will be noted that the evolved hydrogen is approximately 20 per cent too high for a 1/10-scale generation. This

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	Run No.			
	11	12	13	14
Weight of NaBH_4 , lb	10.0	10.0	10.0	10.0
Weight of catalyst ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$), lb	3.20*	3.32*	3.05	3.13*
Volume of river water plus melted ice, gal	56	56	40	25
Initial temperature, F	42	46	35	33
Temperature rise, F	59	59	79	114
Complete reaction time, min	91	77	86	10
Per cent reaction	100	100	100	100
Hydrogen evolved, cu ft	360	361	355	427**
Water vapor evolved, cu ft	10	13	13	26

* Based on the analysis of a sample of the catalyst solution.

**Not reliable, possibly as a result of the capacity of the meter being exceeded.

inaccuracy is possibly due to an inaccuracy in the gas meter used to measure the volume of hydrogen generated. This unit is rated at 30 cu ft per min; this rate was exceeded rather significantly during the last 5 min of the run and possibly, as a result, an erroneous reading was obtained.

These unexpected results prompted a re-check of all of the data, to the extent possible. Since no errors were noted, it appears that, at present, there is no definite explanation for the above-indicated discrepancy.

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Run 14-A was subsequently carried out as a scaled-down version of Run 14; it was conducted in the same open-top glass reaction vessel that previously was used for Runs 1 and 2 (as described in our letter report dated April 14, 1959). The progress of the reaction was followed by recording the temperature rise and also visually noting the time when the reaction ceased. The reaction was almost violent at first, and was complete in 15 minutes. A generation time of 17 minutes is predicted by calculations using the rate equation evolved previously for this hydrolysis reaction; a rapid mixing of the catalyst throughout the system is assumed in these calculations. Such agreement of the experimental with the calculated generation time for Run 14-A (and also for Run 13) suggests that a minimum depth of solution exists for which mixing is no longer rate-controlling.

Since the limited solubility of cobaltous chloride fixes the maximum amount of catalyst that can be added practically in solution, it appeared desirable to check the possibility of adding catalyst as solid crystals. Runs 4-A and 4-B were therefore conducted to check the feasibility of dropping solid catalyst into the borohydride solution.

Runs 4-A and 4-B were carried out under conditions similar to those of small-scale Run 4, which had a reaction time of 42 minutes. In each case, the initial temperature, volume of water, and quantities of the reactants were the same. In Run 4-A, the catalyst was added as lumps of crystals at the top and center of the solution.

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In Run 4-B, crystals of the catalyst were enclosed in a weighted paper capsule made from several thicknesses of paper towel; this arrangement was intended to provide for release of the catalyst particles near or slightly above the bottom of the reactor.

Run 4-A started violently but quickly slowed down. The reaction time was slightly more than 1 hour. In Run 4-B, the paper capsule did not disintegrate as desired. Attempts to puncture the capsule at various times throughout the reaction period in order to release the catalyst were only partially successful. The reaction was somewhat localized around the openings punctured in the capsule wall and continued for more than 3 hours.

These tests indicate that the catalyst when added in the solid form is probably much less effective than when added as a solution. Data for these and the control test are summarized below:

Run No.	Initial	ΔT , F	Total Generation Time, min	Remarks
	Temperature, T_o , F			
4	68	50	42	Solution of catalyst added at center of the reaction pool.
4-A	68	49	66	Catalyst added as lumps. Immediate reaction. Black compound formed (Co_2B); reached bottom of reactor in 6 min, 20 sec.
4-B	68	48	195	Paper towel capsule did not disintegrate as expected. Reaction localized to some extent around holes made in capsule.

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During June, it is planned to investigate further the effect of solution depth on catalyst mixing and on total generation time. The depth effect will be studied in small-scale tests under conditions equivalent to those of Run 14. A 1/5-scale test, equivalent to 1/10-scale Run 14, will then be carried out at about 34 F.

The total appropriation under this Task Order was \$62,579. As of June 1, 1959, the unexpended balance was approximately \$6,400.

Sincerely,

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